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IS 4448 (1994): Benzoic Acid, Food Grade [FAD 8: Food Additives]



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बेंजोइक अम्ल, खाद्य ग्रेड — विशिष्ट

(पहला पुनरीक्षण)

Indian Standard

**BENZOIC ACID, FOOD GRADE —
SPECIFICATION**

(*First Revision*)

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BUREAU OF INDIAN STANDARDS
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**AMENDMENT NO. 1 FEBRUARY 2005
TO
IS 4448 : 1994 BENZOIC ACID, FOOD GRADE —
SPECIFICATION**

(First Revision)

[Page 1, Table 1, Sl No. (i), col 2] — Substitute 'Purity, as $C_7H_6O_2$, percent by mass, on dry basis. Min' for 'Purity, as $C_7H_6O_2$, percent by mass, Min'.

[Page 1, Table 1, Sl No. (ii), col 3] — Substitute '121 – 123°C' for '121.5 – 123.5°C'.

(FAD 8)

Reprography Unit, BIS, New Delhi, India

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division council.

With the increased production of processed foods, manufacturers have started adding a large number of substances, generally in small quantities, to improve appearance, flavour, texture or storage properties of the processed foods. As certain impurities in these substances have been found to be harmful, it is necessary to have a strict quality control of these food additives. A series of standards is, therefore, being prepared by this Bureau to cover purity and identification of these substances. It is hoped that these standards would help in checking purity which requires to be checked at the stage of manufacture, for it is extremely difficult (and in many cases impossible) to detect the impurity once these substances have been added to the processed foods. Besides, these standards are intended to guide the indigenous manufacturers in making their product conform to specification that are accepted by scientists, health authorities and international bodies.

Benzoic acid is bacteriostatic and bacteriocidal agent and preservative for yeast and mould. It is widely used as antimicrobial agent.

Benzoic acid in free state is widely distributed in nature. Gum benzoic from styrax benzoin. Acaroid resin from Xanthorrhoea Hagilis, scent gland of beaver, the bark of wild black cherry tree, ripe cloves and oil of anise seed contain benzoic acid.

Benzoic acid widely used as food preservative, is permitted under the *Prevention of Food Adulteration Rules*, 1955, as well as *Fruit Products Order*, 1955. It is also included in food chemical codex and also falls under IP and BP and USP.

Chemical names and Formula The recognized chemical names are benzoic acid; benzenecarboxylic acid; and phenyl carboxylic acid. Empirical formula of benzoic acid is $C_7H_6O_2$. Its molecular weight is 122.12. Structural formula of benzoic acid is:

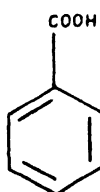


Fig. 1 Structural Formula

This standard was first published in 1967. It is being revised to take into consideration the requirements given in following International as well as EEC Standards as well as the practices being followed in trade.

FAO Food and Nutrition Paper No. 4—Specification for identity and purity of thickening agents, anticaking agents, antimicrobials, antioxidants and emulsifiers; published by the joint FAO/WHO Expert Committee on Food Additives.

Food Chemical Codex, 1981 Pub. National Academy of Sciences and National Research Council, Washington DC, USA.

Council Directive 65/66/EEC of 26 January 1965 laying down specific criteria of purity for preservatives authorised for use in foodstuffs intended for human consumption.

In, this revision solubility has been brought under description and as given in Note under 3.1, it is intended only as information regarding approximate solubility and is not to be considered as a quality requirement. The requirements for polycyclic acid has been deleted as this aspect is already covered under melting range. The requirement for lead has been substituted by heavy metals and a requirement for loss on drying has been included.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2 : 1960 'Rounding of numerical values (revised)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

BENZOIC ACID, FOOD GRADE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for benzoic acid for use as a food preservative.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard.

IS No.	Title
1070 : 1992	Reagent grade water (<i>third revision</i>)
1699 : 1994	Methods of Sampling and test for food colours (<i>second revision</i>)

3 REQUIREMENTS

3.1 Description

Benzoic acid shall be in the form of white crystals, scales or needles. It shall have a faint characteristic odour. Benzoic acid is slightly soluble in water and freely soluble in chloroform and 95 percent ethanol.

NOTE —The solubility is intended only as information regarding approximate solubility and is not to be considered as a quality requirement and is of minor significance as a means of identification or determination of purity and dependence must be placed on other specifications.

3.2 Identification Tests

3.2.1 Reaction with Ferric Chloride

Add 0.1 g of calcium carbonate and 5 ml of water to 0.1 g of the material. Heat gently and filter. To the filtrate, add 4.5 percent aqueous solution of ferric chloride. A buff coloured precipitate is formed.

3.2.2 Place a pinch of the sample in a dry test tube. Wrap the test tube about 4 cm from the bottom with moistened filter paper. Heat the test tube over a low flame. Benzoic acid sublimes and crystals deposit in the colder part of the test tube leaving no residue at the bottom.

3.3 The material shall also conform to the requirements given in Table 1.

Table 1 Requirements For Benzoic Acid
(Clause 3.3)

Sl No.	Characteristics	Requirements	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Purity, as $C_7H_6O_2$, percent by mass, <i>Min</i>	99.5	Annex A
ii)	Melting range	121.5°—123.5°C	Annex B
iii)	Sulphated ash, percent by mass, <i>Max</i>	0.05	Annex C
iv)	Readily carbonizable substances	To conform to test	Annex D
v)	Readily oxidizable substances	To conform to test	Annex E
vi)	Loss on drying (for 3 hours over sulphuric acid or silica gel at ambient temperature in a desiccator) percent by mass, <i>Max</i>	0.5	—
vii)	Chlorinated organic compounds	To conform to test	Annex F
viii)	Arsenic (as As), mg/kg, <i>Max</i>	3.0	15 of IS 1699 : 1994
ix)	Heavy metals mg/kg, <i>Max</i>	10	Annex G

4 PACKING AND MARKING

4.1 Packing

The material shall be filled in amber coloured glass containers, or any other well-closed containers, or suitable bag with inner lining of food grade material, with as little air space as possible. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

4.2 Marking

4.2.1 Each container shall be legibly and indelibly marked with the following information:

- (a) Name of the material, including the words 'Food Grade';
- (b) Source of manufacture;
- (c) Date of manufacture;
- (d) Minimum net contents;
- (e) Batch or code number; and
- (f) Any other requirements as given under the Standards of *Weights and Measures (Packaged Commodities) Rules, 1977/Prevention of Food Adulteration Rules, 1955*.

4.2.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

4.2.2.1 The use of the Standard Mark is governed by the Provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

Representative samples of the material shall be drawn according to the method prescribed in 4 of IS 1699 : 1994.

6 TESTS

6.1 Tests shall be carried out by the methods as specified in 3.2 and col 4 of Table 1.

6.2 Quality of Reagents

Unless otherwise, specified pure chemicals and distilled water (see IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the experimental results.

ANNEX A

[Table 1, Item (i)]

DETERMINATION OF PURITY

A-1 REAGENTS

A-1.1 Phenol Red Solution

Warm 0.05 g of phenol red [phenolsulphonphthalein ($C_{19}H_{14}O_5S$)] with 2.85 ml of 0.05 N sodium hydroxide and 5 ml of 90 percent ethanol; after solution is effected, add a sufficient quantity of 20 percent ethanol to produce 250 ml.

A-1.2 Standard Sodium Hydroxide Solution — 0.5 N.

A-1.3 Phenolphthalein Indicator

Dissolve 0.2 g of phenolphthalein ($C_{20}H_{14}O_4$) in 60 ml of 90 percent ethanol and add a sufficient quantity of water to produce 100 ml.

A-2 PROCEDURE

Weigh 2.5 g of benzoic acid to the accuracy of 0.1 mg which has been previously dried over concentrated sulphuric acid or over silica gel for 3 hours and dissolve in 15 ml of warm ethanol previously neutralized using phenol red. Add 20 ml of water and titrate with standard sodium hydroxide solution, using phenolphthalein as indicator.

A-3 CALCULATION

A-3.1 Calculate the percentage of $C_7H_6O_2$ by the formula given below:

Each ml of 0.5 N sodium hydroxide, is equivalent to 0.061 06 g of $C_7H_6O_2$.

ANNEX B

[Table 1, Item (ii)]

DETERMINATION OF MELTING RANGE

B-1 APPARATUS

B-1.1 Capillary Tube

Thickness of wall 0.10 to 0.15 mm; internal diameter 0.9 to 1.1 mm.

B-1.2 Melting Point Apparatus

Provided with an appropriate liquid like paraffin or silicone oil and a stirring device, and fitted with an auxiliary thermometer.

B-2 PROCEDURE

B-2.1 Spread a small quantity of the finely powdered material in a thin layer and dry at a temperature below its melting temperature, or in a vacuum desiccator over sulphuric acid for 24 hours.

B-2.2 Transfer a quantity of the dried powder to a dry capillary tube and pack the powder by gently tapping the tube on a hard surface so as to form a tightly-packed column 2 to 4 mm high. Attach the capillary tube with its contents to a standard thermometer so that the closed end is at the level of the middle of the bulb, and heat in the melting point apparatus, regulating the rise of temperature during the first period to 3°C per minute. When the temperature reaches to 116.5°C the heating of the apparatus is adjusted, the rate of rise of temperature should be 1°C to 2°C per minute.

B-2.3 Take the reading of the temperature at which the material is observed to form droplets against the side of the tube, and of the temperature at which it is completely melted, as indicated by the formation of a definite meniscus.

B-2.4 To the temperature reading, apply the emergent-stem correction, as follows:

Before starting the determination of the melting range, attach the auxiliary thermometer so that the bulb touches the standard thermometer at a point midway between the graduation for the expected melting range and the surface of the heating material. When the substance has melted, read the temperature on the auxiliary thermometer. Calculate the correction to be added to the temperature reading of the standard thermometer from the following formula:

$$0.00016 N (T - t)$$

where

N = the number of degrees of the scale of the standard thermometer between the surface of the heating material and the level of the mercury.

T = the temperature reading of the standard thermometer, and

t = the temperature reading of the auxiliary thermometer

B-3 INTERPRETATION OF RESULTS

B-3.1 The statement melting range, 121.5° to 123.5°C means that the corrected temperature at which the material is observed to form droplets shall be at least 121.5°C and that the material shall be completely melted at the corrected temperature, that is, 123.5°C.

ANNEX C

[Table 1, Item (iii)]

DETERMINATION OF SULPHATED ASH

C-1 PROCEDURE

C-1.1 Weigh accurately 1.000 to 2.000g of the material in a tared crucible. Ignite until thoroughly charred. Cool, then moisten the residue with one millilitre of sulphuric acid, and cautiously ignite

until the carbon is completely consumed. Conduct the ignition in a place protected from air currents, and use as low a temperature as possible to effect the combustion of the carbon. When the carbon has completely disappeared, cool the crucible in a desiccator and weigh. Note down the weight as ash.

ANNEX D

[Table 1, Item (iv)]

TEST FOR READILY CARBONIZABLE SUBSTANCES

D-1 REAGENTS

D-1.1 Sulphuric Acid — 94.5 to 95.5 percent.

D-1.2 Matching Fluid

Composed of 0.2 ml of cobalt chloride solution (see D-1.2.1), 0.3 ml of ferric chloride solution (see D-1.2.2), 0.1 ml of cupric sulphate (see D-1.2.3) and 4.4 ml of water.

D-1.2.1 Cobalt Chloride Solution

Dissolve about 65 g of cobalt chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$) in enough of a mixture of 25 ml of hydrochloric acid and 975 ml of water to make 1 000 ml. Place exactly 5 ml of this solution in a 250-ml iodine flask, add 5 ml of 3 percent hydrogen peroxide and 15 ml of 20 percent sodium hydroxide solution. Boil for 10 minutes, cool, and add 2 g of potassium iodide and 20 ml of 25 percent sulphuric acid. When the precipitate has dissolved, titrate the liberated iodine with 0.1 N sodium thiosulphate solution adding starch. Each ml of 0.1 N sodium thiosulphate is equivalent to 23.8 mg of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$. Adjust the final volume of the solution by adding enough of the mixture of hydrochloric acid and water to make such milliliter contain 59.5 mg of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$.

D-1.2.2 Ferric Chloride Solution

Dissolve 55 g of ferric chloride in enough of a mixture of 25 ml of hydrochloric acid and 975 ml of

water to make 1 000 ml. Place 10 ml of this solution in a 250-ml iodine flask, add 15 ml of water and 3 g of potassium iodide and allow the mixture to stand for 15 minutes. Dilute with 100 ml of water, and titrate the liberated iodine with 0.1 N sodium thiosulphate solution, adding starch. Each millilitre of 0.1 N sodium thiosulphate is equivalent to 27.03 mg of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$. Adjust the final volume of the solution by addition of enough of the mixture of hydrochloric acid and water to make each millilitre contain 45.0 mg of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$.

D-1.2.3 Cupric Sulphate Solution

Dissolve about 65 g of cupric sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in enough of a mixture of 25 ml of hydrochloric acid and 975 ml of water to make 1 000 ml. Place 10.0 ml of this solution in a 250-ml iodine flask and add 40 ml of water, 4 ml of acetic acid and 3 g of potassium iodide. Titrate the liberated iodine with 0.1 N sodium thiosulphate adding starch. Each millilitre of 0.1 N sodium thiosulphate is equivalent to 24.97 mg of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$. Adjust the final volume of the solution by addition of enough of the mixture of hydrochloric acid and water to make each millilitre contain 62.4 mg of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.

D-2 PROCEDURE

D-2.1 Dissolve 0.500 g of benzoic acid in 5 ml of sulphuric acid; the solution shall not have deeper colour than the matching fluid.

ANNEX E

[Table 1, Item (v)]

TEST FOR READILY OXIDIZABLE SUBSTANCES

E-1 REAGENTS

E-1.1 Sulphuric Acid — 94.5 to 95.5 percent.

E-1.2 Standard Potassium Permanganate Solution — 0.1 N.

E-2 PROCEDURE

E-2.1 Add 1.5 ml of sulphuric acid to 100 ml of

water, heat to boiling and add standard potassium permanganate, solution in drops, until the pink colour persists for 30 seconds. Dissolve 1.000 g of benzoic acid in the heated solution, and titrate with standard potassium permanganate solution to a pink colour that persists for 30 seconds. The amount of 0.1 N standard potassium permanganate solution used shall not exceed 0.5 ml.

ANNEX F

[Table 1, *Item* (vii)]

TEST FOR CHLORINATED ORGANIC COMPOUNDS

F-1 REAGENTS

F-1.1 Sodium Hydroxide Solution — 0.1 N.**F-1.2 Concentrated Nitric Acid** — 69.0 to 71.0 percent.**F-1.3 Calcium Carbonate****F-1.4 Nitric Acid Dilute**

Mix 190 ml of 50 percent nitric acid with sufficient water to make up 1 000 ml.

F-1.5 Silver Nitrate Solution — 0.1 N.**F-1.6 Standard Hydrochloric Acid** — 0.1 N.

F-2 PROCEDURE

F-2.1 Dissolve 0.25 g of benzoic acid in 10 ml of sodium hydroxide solution. Acidify with concentrated nitric acid and filter off the precipitate. Mix the precipitate with 0.5 g of calcium carbonate, dry the mixture and then ignite. Take up the ignited residue in 20 ml of dilute nitric acid and filter. Mix the solution with 0.5 ml of silver nitrate solution. The turbidity shall be not more than that obtained in a similar volume by addition of 0.5 ml of silver nitrate solution and 0.05 ml of 0.1 N standard hydrochloric acid.

ANNEX G

[Table 1, *Item* (ix)]

TEST FOR HEAVY METALS

G-1 REAGENTS

G-1.1 Ammonia Solution

Dilute 400 ml of 28 percent ammonium hydroxide to 1 000 ml with water.

G-1.2 Acetic Acid Solution — 6 percent (m/v)**G-1.3 Hydrochloric Acid** — 10 percent**G-1.4 Lead Nitrate Stock Solution**Dissolve 159.8 mg of lead nitrate $\text{Pb}(\text{NO}_3)_2$ in 100 ml of water containing 1 ml of nitric acid, then dilute with water to 1 000 ml and mix. This solution should be prepared and stored in glass containers which are free from lead salts.**G-1.5 Standard Lead Solution**

On the day of use dilute 10 ml of lead nitrate stock solution, accurately measured, with water to 100 ml. Each ml of the solution so prepared contains the equivalent of 10 microgram of lead ion (Pb).

G-1.6 Hydrogen SulphideA saturated solution of hydrogen sulphide made by passing H_2S in cold water.

G-2 PROCEDURE

G-2.1 Solution A

Take 2 ml of the standard lead solution in a 50-ml Nessler tube and add water to make 25 ml. Adjust the pH to between 3.0 and 4.0 (pH indicator paper) by the addition of diluted acetic acid or ammonia solution. Dilute with water to 40 ml and mix.

G-2.2 Solution B

Volatilize 2 g of benzoic acid over a low flame. To the residue add 2 ml of nitric acid and about 10 mg of sodium carbonate and evaporate to dryness on a steam bath. Dissolve the residue in a mixture of 1 ml of diluted acetic acid and 24 ml of water. Take 25 ml of the above solution in a 50-ml colour comparison tube that matches the one used for solution A and adjust the pH to between 3.0 and 4.0 (pH indicator paper) by the addition of dilute acetic acid solution or ammonia solution. Dilute to 40 ml with water and mix.

G-2.3 To each tube add 10 ml of freshly prepared hydrogen sulphide solution. Mix, allow to stand for 5 minutes and view down over a white surface. The colour of solution B shall not be darker than that of solution A.

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